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Aerotherm Project 6497 March 1976

FOAM COMPOSITE STRUCTURES

By Chadwick B. Delano and Robert J. Milligan

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for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

FOREWORD

This report presents the work accomplished by Acurex/Aerotherm on NASA Contract NAS2-8189, entitled "Development of Advanced Foam Composite Structures," during the 15 May 1975 to 29 March 1976. The program was sponsored by the Chemical Projects Research Office under Dr. J. A. Parker, Chief, National Aeronautics and Space Administration, Ames Research Center, Moffett Field, California 94035. Mr. William Gilwee acted as Technical Monitor.

This program was conducted in the Nonmetallic Materials Department of Aerotherm Division under the direction of Mr. Chadwick B. Delano. Mr. Delano also acted as principal investigator with Mr. Milan Maximovich and Mr. Robert J. Milligan, Chemists and Mr. Denver Dayton, Senior Fabrication Technician assisting.

ABSTRACT

Previous efforts, NASA CR-137597, demonstrated the need to include fire resistant foams into state-of-the-art aircraft interior paneling to increase passenger safety in aircraft fires. Present efforts were directed toward mechanical and fire testing of panels with foam inclusions. Skinned foam filled honeycomb and PBI structural foams were the two constructions investigated in depth with attention being directed toward weight/performance/cost trade-off.

All of the new panels demonstrated improved performance in fire and some were lighter weight but not as strong as the presently used paneling. These present efforts indicate, however, a good area in which continued efforts should result in improved paneling for passenger safety. In particular the simple partial filling (fire side) of state-of-the-art Nomex honeycomb with fire resistant foams with little sacrifice in weight would result in panels with increased fire resistance. More important may be the retarded rate of toxic gas evolution in the fire due to the protection afforded to the Nomex by the foam.

OBJECTIVE

The objective of this program was to identify, characterize, and define the basic physical, mechanical, thermal and economic properties of two concepts of foam composite paneling for aircraft interiors. An additional objective of this program was to determine how best to adhere a nonflammable decorative skin to the panel.

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TECHNICAL DISCUSSION

SECTION 1

INTRODUCTION

An area of great concern in present-day aircraft is the flammability and offgassing characteristics of the structural materials currently used in aircraft interiors. Even when an aircraft fuselage survives a crash intact, lives may be lost in the subsequent fuel fire, as flames penetrate the cabin and/or gaseous byproducts of combustion overcome the passengers within. A prime objective of NASA is to develop aircraft structures that maintain structural integrity and a viable cabin atmosphere for a minimum of 10 minutes under various fire conditions. Such structures would provide protection during the time required for a fuel fire to burn out or for fire crews to extinguish the flames.

Another area of concern involves fires that originate in the rest rooms of commercial aircraft. Such fires, i.e., in trash receptacles, etc., often remain undetected until they develop into major conflagrations and pose a serious threat to the lives of those aboard.

These stringent performance requirements are not the only considerations that must be taken into account. For a design/material concept to be viable, its economics and weight must be attractive to the prime aircraft manufacturer. In this effort where new paneling concepts were evaluated for improvement over currently used materials, panels in particular require smooth, even surfaces and attractive color schemes. The following report details the efforts by Aerotherm to address these areas utilizing two basic foam paneling concepts.

SECTION 2

CONSTRUCTION AND TESTING OF ADVANCED FOAM COMPOSITE STRUCTURES

2.1 CONSTRUCTION AND TENSILE TESTING OF STATE-OF-THE-ART EPOXY/GLASS SKINNED, NOMEX HONEYCOMB CORE STRUCTURES (NON-FOAM FILLED)

Two piles of F224 prepreg cloth (Hexcel) were cured together to foam an impermeable skin. These skins were bonded to 1 inch (2.54 cm) thick HRH-10 1/8 inch (0.32 cm) Nomex honeycomb core (Hexcel) with a roll-on two-part modified epoxy paste adhesive (Hysol EA 9410). The structure was room temperature cured to prevent a pressure buildup which caused earlier attempts to fabricate state-of-the-art composite structures to collapse from the sides inward as they cooled after cure at elevated temperature.

As the end use of these parts is paneling, their weight is expressed in weight unit area. Thickness will be improved separately. Table I gives the weight per unit area of the component parts and the final panels of the fabricated state-of-the-art composite structures. Weight of the adhesive was determined by difference.

Six 2 inch x 2 inch (5.08 cm x 5.08 cm) samples were subjected to flatwise tensile testing to measure their strength at failure and the type of failure. Three were tested at room temperature (298°K) and three at 160°F (344°K). Table 2 lists the data obtained. The results indicate the inability of the room-temperature cure epoxy adhesive to perform well at elevated temperatures, seen in the difference obtained in the failure modes at RT and 160°F .

2.2 CONSTRUCTION AND TENSILE TESTING OF POLYBENZIMIDAXOLE/GLASS SKINNED, POLYBENZIMIDAZOLE BLOWN FOAM STRUCTURES

Two piles of PBI/116 E-glass prepreg were cured together to foam impermeable skins. These skins were bonded to 1 inch (2.54 cm) thick PBI blown foam with both PBI and PPQ adhesive to form the resultant panels.

Table 3 gives the weight per unit area of the component parts and the final panels of the PBI structures fabricated. The adhesive weight was determined by difference.

Although the PBI panels prepared were, overall, lighter in weight than the Nomex-Epoxy panels above, some of this weight savings came from the use of less adhesive/unit area.

TABLE 1. WEIGHT/UNIT AREA OF SUBASSEMBLIES AND THE ASSEMBLED CONVENTIONAL AIRCRAFT PANEL

Subassembly	Weight/Unit Area	a, 1b/ft ² (kg/m ²
Two (2) F-224 Epoxy/ 120 glass skins, two ply	0.14	(0.69)
HRH-10 Nomex core	0.15	(0.73)
Hysol EA9410 Adhesive	0.11 - 0.19	(0.54 - 0.93)
Final Panels, 1.03 in (2.62 cm) Thick	0.40 - 0.48	(1.95 - 2.34)

TABLE 2. FLATWISE TENSILE RESULTS OF STATE-OF-THE-ART EPOXY/GLASS SKINNED, NOMEX HONEYCOMB CORE STRUCTURES

Temp, °F (°K)	Tensile Strength at Failure, psi (kN/m²)	Point of Failure
RT (298°)	211 (1450)	Core
	208 (1430)	
	223 (1540)	∤
160° (344°)	131 (903)	90% - 95% Adhesive
	124 (855)	
	113 (779)	

TABLE 3. WEIGHT/UNIT AREA OF CONSTITUENTS AND FINAL POLYBENZIMIDAZOLE PANEL

Constituents	Weight/Unit Area, 1b/ft² (kg/m²)	
Two (2) Polybenzimidazole 116-Volan skins, 2 ply	0.11 - 0.12	(0.54 - 0.59)
Polybenzimidazole Blown Foam Core	0.16 - 0.20	(0.78 - 0.98)
PBI, PPQ Adhesives	0.03 - 0.04	(0.15 - 0.20)
Final Panels, 1.08 in (274 cm) Thick	0.30 - 0.36	(1.47 - 1.76)

Twelve 2 inch x 2 inch (5.08 cm x 5.08 cm) samples were subjected to flatwise tensile testing to measure their strength and observe the failures. Six of the samples were bonded with polyphenylquinoxaline adhesive (PPQ) and six with polybenzimidazole (PBI). Half of each was tested at room temperature and the other half at 160°F (344°K). The data is presented in Table 4.

The tensile strengths at failure were found to be consistent with those values which would be expected of PBI foam of that density $(1.8 - 2.3 \text{ lb/ft}^3, 29 - 37 \text{ kg/m}^3)$.

2.3 CONSTRUCTION AND TENSILE TESTING OF KERIMID 601/GLASS SKINNED, P-NASA FOAM FILLED KERIMID/ GLASS HONEYCOMB CORE STRUCTURES

2.3.1 Without FM-34 Adhesive

Kerimid 601/glass honeycomb core, 1 inch (2.54 cm) thick, consisting of 3/8 inch (0.95 cm) cells and having a nominal density of 4 lb/ft³ (64.1 kg/m³) was obtained from Hexcel. This foam core was dipped in a well shaken slurry of ammonium 5-nitro-2-aminobenzenesulfonate (p-NASA) and nitrocellulose in ethyl acetate/toluene. After drying, the coating was ignited by heating to 280°C. The pyrolysis residue completely filled the core. However, this residue had little or no adhesion to the side-walls of the honeycomb core. The residue was then lightly brushed out of the honeycomb to a depth of approximately 1 mm. A coating of BR-34 polyimide primer was then applied to the exposed honeycomb and 1 ply Kerimid 601/116 E-glass skins were bonded to each side.

The weight/unit area of the structures and their constituents is given in Table 5.

The panels are just under the target weight limit of 0.70 lb/ft^2 (3.4 kg/m^2). In order to keep these structures below this weight limit, it was decided to do without the combination FM-34 adhesive/BR-34 primer system and to bond the 1-ply Kerimid skin directly to the core.

Six 2 inch x 2 inch (5.08 cm x 5.08 cm) samples were subjected to flatwise tensile testing to measure their strengths and observe the failures. Half of these samples were tested at room temperature (298° K) and the other half at 160° F (344° K). Table 6 contains the data from these tests. Thus it appears that for the most part the failure occurred between the Kerimid and the Volan A-finished 116 E-glass.

2.3.2 With FM-34 Adhesive

One such structure was prepared. The BR-34 primer was applied in the same manner as described above. A layer of FM-34 adhesive was then placed between the BR-34 primed honeycomb and the skin. The resultant composite's weight breakdown is shown in Table 7.

TABLE 4. FLATWISE TENSILE RESULTS OF POLYBENZIMIDAZOLE BLOWN FOAM COMPOSITE STRUCTURES

Adhesive	Temp °F (°K)		Strength at psi (kN/m²)	Point of Failure
PPQ	RT (298)	14.5	(100.0)	100% Foam
		11.0	(75.8)	100% Foam
	Market Continues of the	19.3	(133.1)	85% Foam/15% Adhesive
PPQ	160 (344)	7.8	(53.8)	95% Foam/5% Adhesive
		12.6	(86.9)	100% Foam
		9.5	(65.5)	100% Foam
PBI	RT (298)	8.5	(58.6)	25% Foam/75% Adhesive
		20.5	(141.3)	95% Foam/5% Adhesive
		12.8	(88.3)	100% Foam
PBI	160 (344)	7.3	(50.3)	85% Foam/15% Adhesive
		12.3	(84.8)	75% Foam/25% Adhesive
		13.0	(89.6)	90% Foam/10% Adhesive

TABLE 5. WEIGHT/UNIT AREA OF CONSTITUENTS AND FINAL KERIMID/GLASS SKINNED P-NASA FOAM FILLED, KERIMID/GLASS HONEYCOMB STRUCTURE

Constituents	Weight/Unit Area,	lb/ft² kg/m²)
Two (2) Kerimid 60/116 E-glass skins, 1 ply	0.068 - 0.080	(0.33 - 0.39)
3/8 Inch Kerimid, 601/glass honeycomb core	0.31 - 0.32	(1.51 - 1.56)
p-NASA foam*	0.15 - 0.16	(0.73 - 0.78)
BR-34 primer** (cured)	0.07 - 0.12	(0.34 - 0.59)
Composite panels	0.60 - 0.68	(0.92 - 3.32)
		

^{*} Weight of p-NASA foam determined by subtraction; honeycomb + p-NASA — honeycomb.

Weight of BR-34 primer determined by difference.

TABLE 6. FLATWISE TENSILE RESULTS OF P-NASA FOAM FILLED KERIMID/GLASS HONEYCOMB COMPOSITE STRUCTURES

Temperature °F (°K)	Tensile Strength at Failure psi (kN/m²)	Point of Failure Kerimid/Skin glass Primer	
RT (298°)	113 (779)	85%	15%
	75 (517)	50%	50%
	113 (779)	85%	15%
160 (244°)	69 (476)	85%	15%
	86 (593)	80%	20%
	88 (607)	85%	15%

TABLE 7. WEIGHT/UNIT AREA OF CONSTITUENT AND FINAL KERIMID 601/GLASS SKINNED, P-NASA FOAM FILLED, KERIMID 601/GLASS HONEYCOMB STRUCTURE BONDED WITH FM-34 ADHESIVE USING BR-34 PRIMER

Constituents	Weight/Unit Area lb/ft² (kg/m²)
Two (2) Kerimid 116E Glass Skins, 1 Ply	0.071 (0.35)
3/8" Kerimid 601 Glass Honeycomb Core	0.36 (1.76)
p-NASA Foam	0.15 (0.73)
BR-34 Primer/FM-34 Adhesive*	0.13 (0.63)
Composite Panel	0.71 (3.46)
*0.03 lb/ft² x 2	

Three flatwise tensile specimens were cut from this panel and tested to see whether the use of the FM-34 adhesive improved the strength to any significant degree. Table 8 gives the results obtained.

The type of failures observed in Table 6 with the Kerimid debonding from the glass were not observed when the FM-34 adhesive was used. The failures were primarily between the Kerimid/glass core and the FM-34 adhesive although to a small amount ~5 percent it appeared as if debonding occurred between the Kerimid and the glass in the core. The results indicate a 30 percent increase in tensile strength at room temperature was observed by the use of the adhesive.

2.4 CONSTRUCTION AND TENSILE TESTING OF KERIMID/GLASS SKINNED, P-NASA FOAM FILLED NOMEX HONEYCOMB CORE STRUCTURES USING FM-34 ADHESIVE AND BR-34 PRIMER

To reduce the weight of the p-NASA foam package, Nomex honeycomb core (1.8 lb/ft³, 28.8 kg/m³) was substituted for the Kerimid/glass core used above. The resultant filled honeycomb showed a propensity for warping and or internally exploding if the honeycomb was not adequately ventilated and held level during the thermal decomposition of the p-NASA/nitrocelluose coating. The resulting filled honeycomb structures were brushed to expose approximately 1 mm of honeycomb, and using BR-34 primer and FM-34 adhesive, Kerimid/Volan A finished 116 E-glass skins were bonded to both sides. Heavy FM-34 adhesive (0.135 lb/ft²) was used on the structure. If the light (0.030 lb/ft²) material were used, the structure would be, on the average 0.21 lb/ft² lighter. Table 9 lists the weight/ unit area of the components, the final structure, and the final idealized structure if the lighter adhesive were used. A net savings in weight was not realized with the Nomex honeycomb due to increased weight of primer required for coating the 1/8 cells of the Nomex honeycomb as opposed to the 3/8 inch cells used in the Kerimid/glass honeycomb.

Table 10 shows the results of flatwise tensile of samples at both 298°K and 344°K. These results indicate that the Nomex core is degraded by the reaction that creates the p-NASA residue. Comparison of these results to Table 2 show that the tensile strength of the p-NASA containing Nomex is a fraction of the unfilled Nomex honeycomb.

2.5 CONSTRUCTION OF KERIMID/GLASS SKINNED P-NASA FOAM FILLED POLYIMIDE HONEYCOMB CORE STRUCTURE

A half inch thick sample of HRH 327 polyimide honeycomb core with 3/8 inch cells and having a nominal density of 4 lb/ft³ (64.1 kg/m³) was coated with a slurry of p-NASA and allowed to dry. The coating was then ignited to give overall a more uniform coating than what was achieved with the Kerimid honeycomb. There appeared to be better adhesion between the walls of the honeycomb cells and the residue. No further tests were carried out on this panel.

TABLE 8. FLATWISE TENSILE RESULTS OF P-NASA FOAM FILLED KERIMID/GLASS HONEYCOMB COMPOSITE STRUCTURES BONDED WITH BR-34 PRIMER AND FM-34 ADHESIVE

Temp °F (°K)	Tensile Strength at Failure, psi (kN/m²)	Point of Failure
RT (298)	141 (970)	55% core/primer 40% primer/adhesive
	149 (1025)	85% core/primer 15% primer/adhesive
160 (344)	88 (605)	50% core/primer 50% primer/adhesivo

TABLE 9. WEIGHT/UNIT AREA OF CONSTITUENTS AND FINAL KERIMID/GLASS SKINNED, P-NASA FOAM FILLED NOMEX HONEYCOMB STRUCTURE BONDED WITH FM-34 ADHESIVE AND BR-34 PRIMER

Constituents	Weight/Unit Area,	lb/ft² (kg/m²)
Two (2) Kerimid 116 Voian finished E-glass skins, 1 ply	0.074	(0.36)
1/8" Nomex Honeycomb core 0.915 in. thick	0.15	(0.73)
p-NASA foam	0.19	(0.93)
Two (2) FM-34 adhesive 112 E-glass	0.27	(1.32)
BR-34 primer	0.25	(1.22)
Final Composite Structure	0.93	(4.54)
Calculated Final Composite structure if lightweight FM-34 adhesive used.	0.72	(3.51)

TABLE 10. FLATWISE TENSILE RESULTS OF P-NASA FOAM FILLED NOMEX HONEYCOMB COMPOSITE STRUCTURES

Temp °F (°K)	Tensile Strength, psi (kN/m²)	Point of Failure
RT (298)	48.5 (335)	50% Core 50% Primer-Core
	30 (205)	100% Core
160 (344)	40 (275)	100% Core
	30 (205)	100% Core

2.6 AMES T-3 TESTING OF SELECTED FOAM COMPOSITE STRUCTURES

Samples of the three foam composite structures described in paragraphs 2.1, 2.2, and 2.3.1 were submitted to NASA-Ames for T-3 testing. These panels, 8 inch (20.3 cm) x 8 inch (20.3 cm) x 1 inch (2.54 cm), were mounted with two thermocouples; one in the center of the structure thickness and the second on the center of the backside as shown in Figure 1. The panels were then placed in the Ames T-3 thermal test facility at area (1), as shown in Figure 2 and exposed to a heat flux resulting from a controlled JP-4 fuel fire. Figures 3 through 5 show the ability of the three composite foams to delay temperature increases to the backside of the structure. The premature termination of the state-of-the-art epoxy skinned Nomex honeycomb structure was due to the fact that the test sample shrank away from its mountings and was consumed in that period of time. The other two composite foam structures maintained their dimensional integrity throughout the entire ten minute test span.

Table 11 provides the time to reach a backside of 300°F for four foam composite structures. The p-NASA filled Karimid honeycomb structure at 0.68 lb/ft² required 270 seconds for the backside temperature to reach 300°F. This is about twice as long as an equivalent weight PBI foam structure and 5 times longer than the state-of-the-art structure which is about 1/2 of the weight.

The p-NASA foam should prove to provide even longer times to reach the 300°F backside temperature if a smaller celled honeycomb is used.

2.7 SMOKE DENSITY AND RESPIRATORY DISTRESS RESULTS

These tests were run for NASA-Ames by the University of San Francisco. The results, listed in Table 12 show the extraordinary lack of particulate matter released by PBI under flaming combustion conditions. The smoke generated by the p-NASA foam filled, Kerimid skinned composite panel is probably due to the flaming combustion of Kerimid 601 resin.

Despite the relatively smaller amount of particulate matter released from the p-NASA Kerimid composite panels as compared to state-of-the-art Nomex honeycomb composite, the panels in test were comparable in the time required to initiate respiratory distress in test animals (white rats). In this respect too, PBI foam was superior.

There appeared to be little difference in smoke generation between the PBI composite foam sample using PPQ adhesive (DD-337-20) and that using PBI adhesive (DD-337-17C). The smoke generated from the former was not tested for respiratory distress.

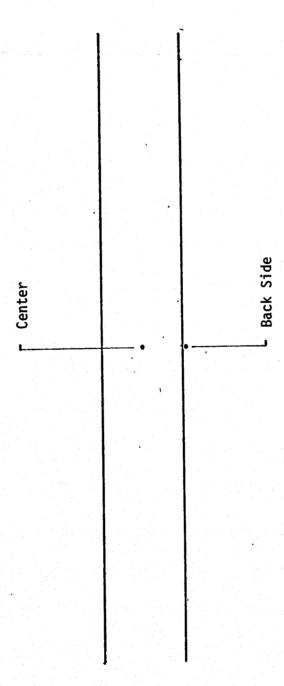


Figure 1. Thermocouple placement, skinned sandwich.

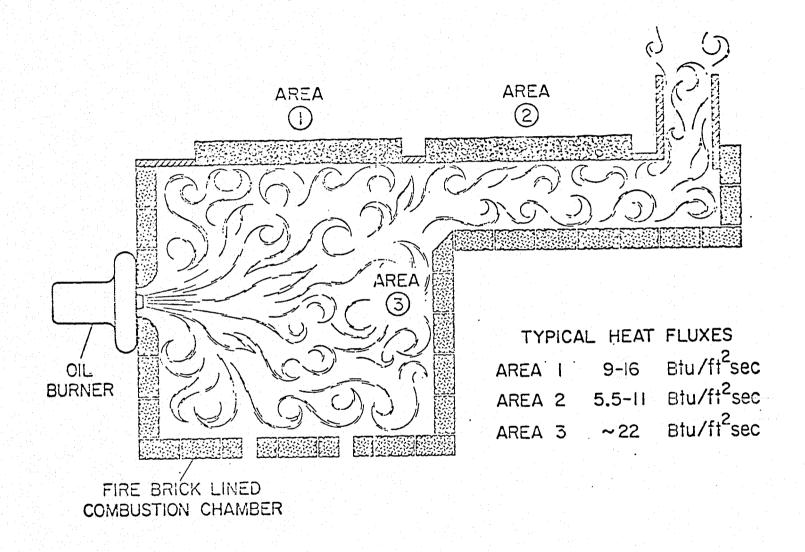


Figure 2. Ames T3 thermal test facility JP-4 fuel.

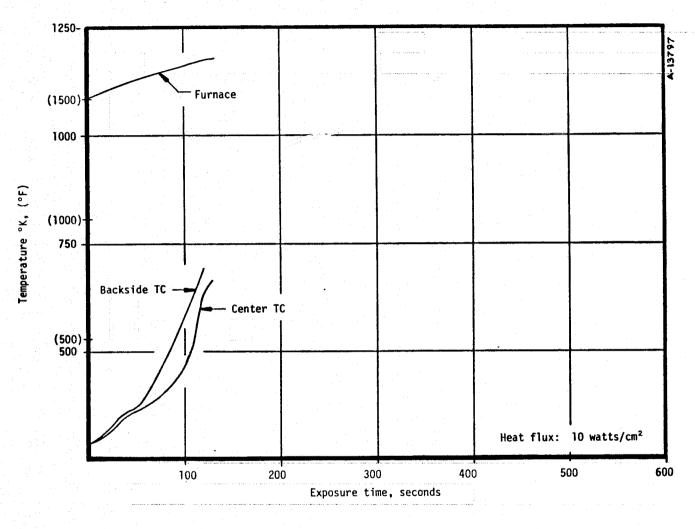


Figure 3. T-3 results — Nomex core — epoxy skin panel.

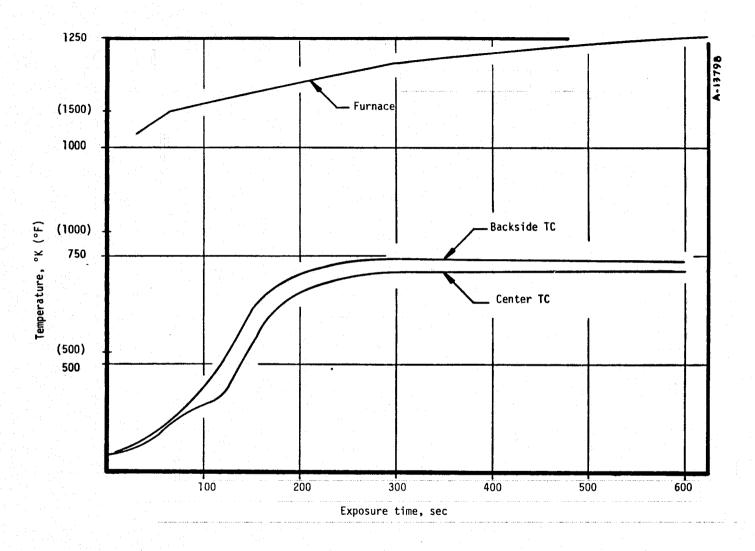


Figure 4. T-3 results — PBI blown foam core, PBI skins.

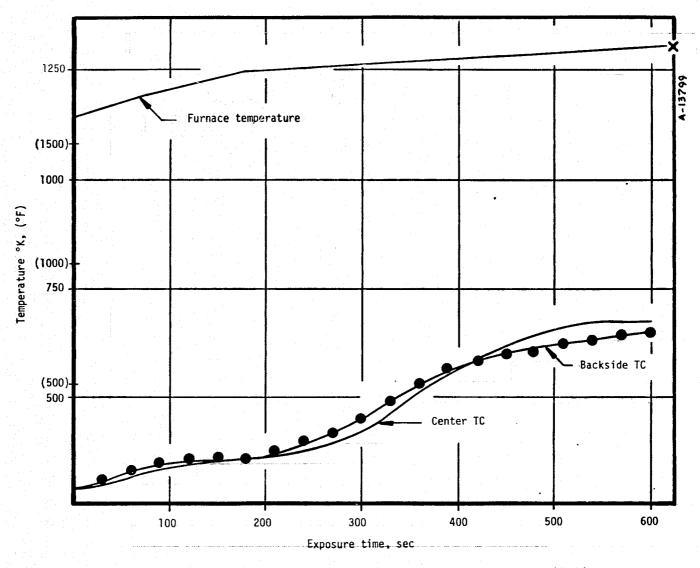


Figure 5. T-3 results - P-NASA foam filled Kerimid honeycomb core, Kerimid skins.

TABLE 11. THERMAL INSULATIVE EFFICIENCY OF VARIOUS COMPOSITE FOAM STRUCTURES

Panel Number	Type Foam	Weight/Unit Area From 1b/ft ² (kg/m ²)	Heat Flux Btu/ft ² /sec (watt/m ² x 10 ⁴)	Furnace Temperature °F (°K)	Time Required for Backside Temp to Reach 300°F (422°K), Sec
DD 337-15A	Nomex Honeycomb	0.40 (1.95)	10 (11.4)	~1600 (1150)	65
DD 337-22	PBI	0.35 (1.70)	10-11 (11.4-12.5)	~1600 (1150)	90
Panel 13*	PBI	0.70 (3.41)	10-11 (11.4-12.5)	~1550 (1120)	110
DD 337-37	P-NASA	0.68 (3.32)	10-13 (11.4-14.7)	~1800 (1250)	270

TABLE 12. SMOKE DENSITY TEST RESULTS ON AIRCRAFT INTERIOR PANELS — FLAMING EXPOSURE, 2.5 W/cm²

0 7 7 130 - 13	Test	S	ample W	eight,	g	Specific	Optical	Density Ds_	Time to Maximum	Time to First Sign of Respiratory
Sample Identification	No.	Initial	Final	Loss	% of Loss	1.5 min	4.0 min	Maximum Dm	(Min)	Distress, (min)
DD 337-15C-1	1	12.4	10.9	1.5	12.0	24.6	33.1	43.1	17.0	7.2
State-of-the-art (2	12.2	10.9	1.3	10.7	3.2	27.3	32.9	10.5	10.0
<pre>} epoxy skinned { Nomex honeycomb</pre>	1	12.1	10.8	1.3	10.7	26.0	35.4	42.2	18.5	
DD 337-15C-2	2	12.1	10.6	1.5	12.4	20.0	28.2	33.0	9.8	7.5
DD 337-39A Kerimid skinned p-NASA	1	19.5	17.6	1.9	9.7	1.7	6.3	14.2	12.0	9.0
DD 337-39B foam filled Kerimid honeycomb	1	10.5	17.5	2.0	10.3	1.5	3.2	12.1	19.0	
DD 337-17C PBI skinned	1	8.5	8.0	0.5	5.9	0.6	0.6	0.8	13.0	19.0
DD 337-20 PBI blown foam	1	9.7	9.0	0.7	7.2	0.6	0.6	0.8	15.5	

SECTION 3

ADDITIONAL MECHANICAL TESTING OF P-NASA FOAM FILLED KERIMID/GLASS SKINNED KERIMID/GLASS HONEYCOMB COMPOSITES

Based on the overall results in paragraph 2, additional mechanical testing on the p-NASA foam filled composite structures were carried out.

3.1 PREPARATION OF THE FOAM SAMPLES

The composite foam samples were prepared in the same manner as the samples submitted to NASA-Ames for the T-3 smoke and respiratory tests.

3.2 COMPRESSIVE TESTING

Eight 2 inch (5.1 cm) x 2 inch (5.1 cm) x 1 inch (2.54 cm) samples were cut from a composite foam panel and subjected to flatwise compression loading according to ASTM C365-57. Four of the specimens were tested at room temperature and the other four at 150°F. Table 13 contain the results of this testing. Little difference in values were noticed between the two temperatures.

3.3 TENSILE TESTING

Eight 2 inch (5.1 cm) x 2 inch (5.1 cm) samples were cut from a panel and subjected to flatwise tensile loading after the outer skins had been bonded to steel blocks with EA 9410 adhesive. The tests were run under the conditions prescribed in ASTM C297-61. Four of the specimens were run at room temperature and the remaining four at 160°F. As before, debonding occurred between the Kerimid and the glass weave of the outer skins. Some debonding was also noted between the Kerimid and the glass of the honeycomb. The results are listed in Table 14.

3.4 CORE SHEAR TESTING

This test was conducted according to ASTM Designation C273-61. The dimensions of core as used was 6.0 inches (152.4 mm) x 2.0 inches (50.8 mm) x 1/2 inch (12.7 mm). The foam core was bonded directly to the fixtures with EA-9410 adhesive. Six specimens were used. Three were tested at room temperature and three at 160°F . The results are in Table 15.

TABLE 13. COMPRESSIVE TESTING OF P-NASA FOAM FILLED COMPOSITE STRUCTURES

Temperature °F (°K)	Compressive Strength At Failure, psi (kN/m²)
RT (298°)	337 (2324) 344 (2372) 354 (2441) 334 (2303)
160° (344°)	333 (2296) 334 (2303) 315 (2172) 360 (2482)

TABLE 14. TENSILE TESTING OF P-NASA FOAM FILLED COMPOSITE STRUCTURES

Temperature	Tensile Strength at Failure			
°F (°K)	psi (kN/m²)			
RT (298°)	161 (1165) 141 (972) 192 (1324) 151 (1041) 146 (1007) 117 (807) 100 (689) 137 (945)			

TABLE 15. SHEAR TEST OF FLAT SANDWICH CORES

Temperature °F (°K)	Ultimate Shear psi (kN/m²)	Type Failure
RT (298°)	293 (2020) 256 (1765) 257 (1772)	Shear Shear Shear
160° (344°)	184 (1269) 241 (1662) 221 (1524)	Shear/adh. 100% Shear Shear/adh.

3.5 FLEXURAL TESTING

Flexural testing was run according to the ASTM C393 and MIL-STD-401A, Section 5.2.4. Six 8.0 inch (203.2 mm) x 2.0 inch (50.8 mm) x 1.0 inch (25.4 mm) samples were cut so that the sample length was parallel to the core ribbon direction. These specimens were then subjected to four point flexural loading as shown in Figure 6. The bottom span was 6 inches and the top span was 2 inches. Three of the specimens were tested at room temperature and the remaining three at 160° F.

Deflection was measured by mounting a dial micrometer at the center underside of the specimen (point a). Table 16 gives the unreduced data from this test while Figures 7 and 8 give the strength/deflection curves.

Failure of all of the specimens was by the same mode. The upper skin debonded from the core and buckled when the honeycomb compressed. Flat narrow plates were placed beneath the load bearing surfaces to prevent crushing at the load points. The results indicated that the failures resulted from bending rather than shear. Test parameters needed to give shear failure for the composite would require that force be within 0.20 inch of the reaction points. Such a test is not possible due to the crushing observed unless stronger skins are used. Interlaminar shear would provide a similar result as the core shear previously reported.

Further, for these specimens the flexural strength with load/deflection curve and the beam bending with load/deflection curve are almost identical. Flexural stiffness of the composite and the modulus of elasticity of the glass skins were calculated and are presented in Table 17.

The value obtained for E is approximately one-fifth the modulus of the resin/E-glass used in the skins and obviously reflects the puckering of the skins into the core.

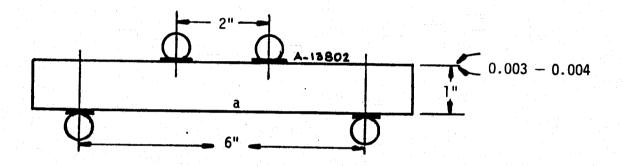


Figure 6. Schematic sketch of composite foam loading for flexural testing.

TABLE 16. DATA OBTAINED FROM FLEXURAL TESTING

Temperature °F (°K)	Maximum Load at Failure, PB lbs (N)	Deflectional at Failure in/in = (m/m)
RT (298)	115 (512) 125 (556) 105 (467)	0.088 0.084 0.082
160° (344)	99 (441) 82 (365) 105 (467)	0.073 0.058 0.080

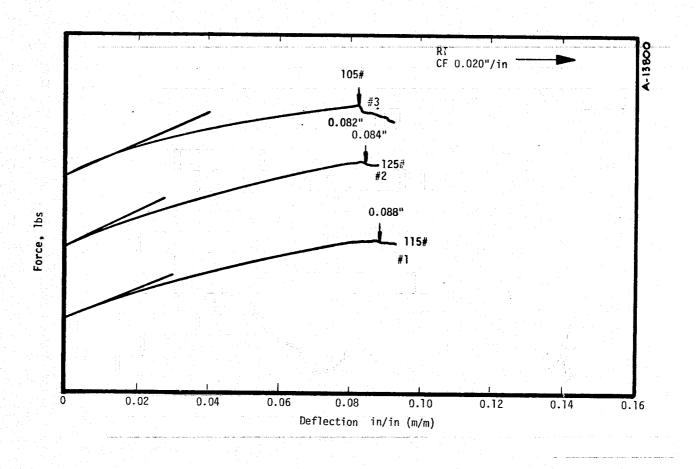


Figure 7. Load deflection curves, p-NASA foam filled composite, RT (298°K).



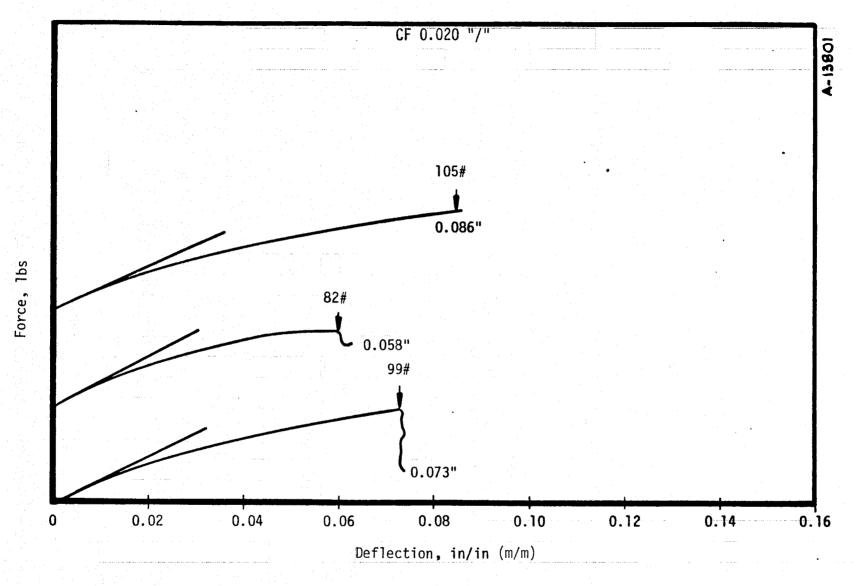


Figure 8. Load deflection curve, p-NASA foam filled composite, 160°F (344°K).

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TABLE 17. CALCULATED FLEXURAL STIFFNESS AND CALCULATED MODULUS OF ELASTIC-ITY OF THE GLASS SKINS FOR P-NASA FOAM FILLED COMPOSITES

Temperature °C (°K)	PB/WB (in-lb/in)	Flexural Stiffness D lb/in² (MN/m²)	Modulus of Elasticity E lb/in ² (GN/m ²)
RT (298)	2300	7100 (48.9)	480,000 (3.31)
	2550	7900 (54.5)	536,000 (3.70)
	2100	6500 (44.8)	440,000 (3.03)
160 (344)	2400	7400 (51.0)	504,000 (3.48)
	2600	8000 (55.2)	546,000 (3.76)
	2450	7600 (52.4)	514,000 (3.54)

SECTION 4

DECORATIVE SURFACES

Two Kerimid/glass skinned, p-NASA foam filled, 3/8 inch Kerimid honeycomb core blocks, 3 inches (76.2 mm) x 6 inches (152.4 mm) x 1 inch (25.4 mm), were coated on both sides with white phenolphthalein polycarbonate films for improved cosmetics. As the Kerimid skin of these panels was quite dimpled, screen effects were carried on flat pieces of Kerimid/glass. Two types of adhesives were to be initially employed, PPQ 401 and a chlorinated epoxy resin system.

4.1 ATTEMPTS TO BOND PHENOLPHTHALEIN POLYCARBONATE TO KERIMID/GLASS SKINS WITH PPQ 401 ADHESIVE

A test sample of Kerimid skin was lightly sanded and solvent wiped. It was then coated with a 16 percent solution of PPQ 401 in m-cresol. After some evaporation of solvent to tackiness, the polycarbonate film was placed on the skin and the assembly cured in a press.

The results indicated that the m-cresol solvent is too compatible with the phenolphthalein polycarbonate for use as an adhesive. As the structure was heated the m-cresol began to dissolve in the polycarbonate. Eventually, when the temperature became high enough to expel this solvent, it left folds and wrinkles in the polycarbonate surface as well as nonbonded areas. Generally these nonbonded areas were accompanied by cracks in the polycarbonate skin.

4.2 BONDING OF PHENOLPHTHALEIN POLYCARBONATE TO KERIMID SKINS WITH AN EPON 828/CHLORENDIC ANHYDRIDE RESIN

The method of preparing the chlorendic anhydride/Epon 828 epoxy resin and of bonding the phenolphthalein polycarbonate film to the Kerimid skins with this adhesive are described in Section 6.3 of Appendix G. Smooth Kerimid surfaces were easily bonded to film and only a minimal amount of debonded areas were apparent. However, the dimpled surface of the composite foam specimen showed numerous air pockets and cracks in the film. A 3 inch (76.2 mm) x 6 inch (152.4 mm) x 1 inch (25.4 mm) sample of this panel was submitted to NASA-Ames.

4.3 BRUSH COATING PHENOLPHTHALEIN POLYCARBONATE FILMS

To circumvent the numerous debonded areas, phenolphthalein polycarbonate solutions with titanium dioxide (rutile) added as an opacifier and filler were prepared and experiments were run to

judge the best way of applying these solutions to swatches of cured Kerimid/glass composite. The results indicated that:

- a. The surface had to be thoroughly sanded
- b. The polycarbonate solution had to be applied in several thin coats and partial but not complete drying of each coat had to occur prior to overcoating.

A 3 inch $(76.2 \text{ mm}) \times 6$ inch $(152.4 \text{ mm}) \times 1$ inch (25.4 mm) sample was coated both sides. This panel was submitted to NASA-Ames.

SECTION 5

APPROXIMATE MATERIAL AND PROCESSING COSTS

Material and processing costs are described for both the PBI blown foam panels and the various p-NASA foam panels prepared. No attempt was made to calculate a weight-penalty cost.

5.1 POLYBENZIMIDAZOLE (PBI) BLOWN FOAM PANELS

5.1.1 PBI Costs

The key to the material cost of PBI blown foam panels is the price and availability of 3,3', 4,4'-tetraminobiphenyl. Currently this chemical, a monomeric constituent of both PBI and PPQ, is unavailable commercially. If required Aerotherm has developed a synthesis of this compound, which when scaled up, should produce batches of the material at \$25 to \$50/lb (\$55 to \$110/Kg) based upon a moderate consumption of 10,000 lb/annum.

5.1.2 Processing Steps

Figure 9 shows the steps required in producing a composite PBI panel starting with the required raw materials. At the present laboratory scale for a 1000 board feet of the skinned PBI, it is estimated that the cost would be about \$140/board foot. Economics of scale would be realized with large markets to reduce this cost accordingly.

5.2 P-NASA FOAM FILLED HONEYCOMB PANELS

All materials for the construction of these composites are commercially available except for the Kerimid honeycomb. Preparation of the p-NASA foam filled panels involved the use of three different honeycomb cores and an experimentation with various combinations of FM-34 adhesive and BR-34 primer. Costing of variations follows.

5.2.1 Processing Steps

Figure 10 shows the steps required in producing a composite p-NASA panel starting with the basic chemicals and other purchased materials. Limitation to the size panels that can be prepared are due to the limitation in producing Kerimid skins although the uniform coating of the form with p-NASA salt may also be a factor.

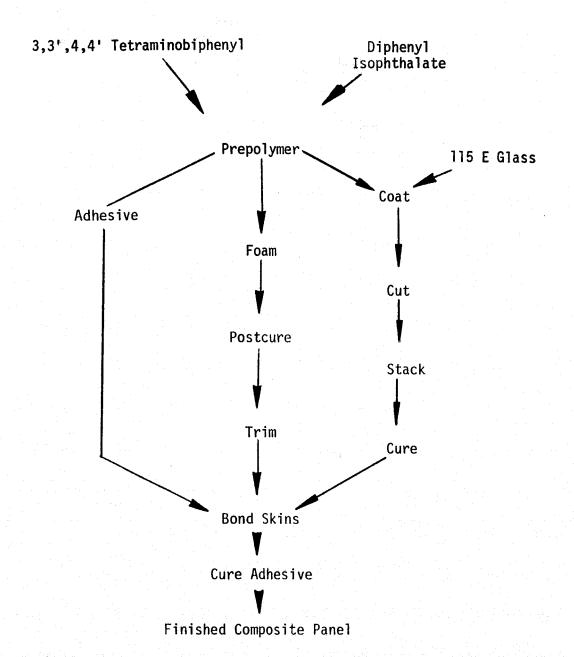


Figure 9. Processing steps involved in the preparation of polybenzimidazole (PBI) blown foam composite panels.

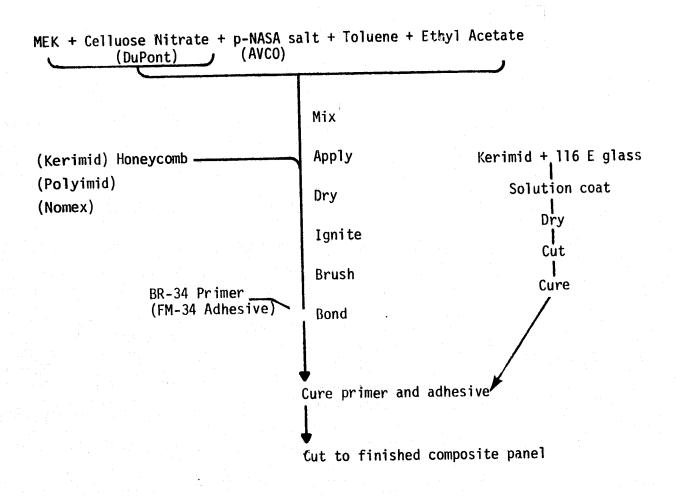


Figure 10. Processing steps required in the preparation of p-NASA foam filled Kerimid honeycomb core composites.

5.2.2 Processing and Material Costs

5.2.2.1 Kerimid/Glass Honeycomb Core

The 3/8 inch core has a nominal density of 4 lb/ft³ (64 kg/m³). Thus, a one inch (2.54 cm) thick slab weighs 0.33 lb/ft² (1.61 kg/m²). The cost for this special order item was \$75.21/ft² (\$800/m²).

5.2.2.2 The p-NASA Foam

The constituents for the coating that is fired to produce the p-NASA foam are the following:

- Celluse nitrate (5 second type), 18 percent solids in methylethylketone
- (MEK); 41 percent
- p-NASA salt (ammonium 2-amino-5-nitrobenzenesulfonate); 23 percent
- Toluene; 18 percent
- Ethyl acetate; 18 percent.

Celluose nitrate is purchased from Du Pont as 70 percent solids in ethanol, i-propyl alcohol or n-butanol; minimum order 200 lbs of solids. One pays \$0.94/lb for the 5 to 6 second variety plus approximately \$0.20/lb for the alcohol solvent. To blend all of it with methyl ethyl ketone requires 825 lbs of the ketone to form the 18 percent solids mixture. Methyl ethyl ketone, MEK, in this quantity is purchased for \$0.18/lb.*

A 621 1b batch of p-NASA salt at \$7.50/1b requires 489 1bs of toluene (\$0.09/1b)* and 489 1bs of ethyl acetate (#0.22/1b)*. This mixture would cost about \$1.90/1b for the chemicals alone.

The blending operation requires shaking the mixture with ceramic balls to provide — at least for a finite period of time — a uniform suspension of the p-NASA salt. The labor required for blending is included in the labor required for dunking the honeycomb, drying and finally foaming. Cost/ ft^2 of paneling, figuring a 10 to 20 percent waste of p-NASA slurry and an additional ~15 percent scrap factor of foamed core is about \$40 to \$100/ft^2. Approximately 25 lbs of the p-NASA slurry would be consumed in the operation.

5.2.2.3 Production of the Kerimid 601 116 E-Glass Skins

Including labor, the cost for the Kerimid skins should not exceed \sim \$5/ft² (\$53.80/m²).

Prices for these solvents were obtained or derived from prices quoted by Chemical Marketing Reporter, Vol., No. 21, November 24, 1975, Schnell Publishing Company, New York, N.Y.

5.2.2.4 Panel Cost

It is estimated that the Kerimid/glass honeycomb filled with the p-NASA and finished with Kerimid/glass skins will cost about $126/ft^2$. Economies of scale would reduce this cost accordingly.

SECTION 6 CONCLUSIONS AND RECOMMENDATIONS

Table 18 compares the three different composite systems.

TABLE 18. EVALUATION OF COMPOSITE PANELS

	Rating ^a				
	Most Desi	rab	le → Lea	ast	Desirable
Weight/Unit Area	PBI	~	STA	>>	p-NASA
Tensile Strength	STA	>	p-NASA	>>	PBI
Thermal Insulative Efficiency	p-NASA	>>	PBI	>	STA
Smoke Density	PBI	>	p-NASA	>	STA
Respiratory Distress	PBI	>>	p-NASA	~	STA
Cost	STA	>	p-NASA	≅	PBI
aPBI — PBI/glass skinned, PBI foam core STA — Epoxy/glass skins, Nomex core p-NASA — p-NASA foam filled Kerimid/glass honeycomb core, Kerimid/glass skins					

Table 18 shows that the state-of-the-art panels cost the least and have the highest strength but at the same time are the least desirable in a fire environment. It is recommended that the major research emphasis be placed in finding economical nonburning materials which can be used for foam filling of the core or for the core itself.

APPENDIX A

PRELIMINARY PROCESS SPECIFICATIONS FOR THE PREPARATION OF EPOXY SKINNED NOMEX HONEYCOMB CORE COMPOSITE

- 1.0 SCOPE
- 1.1 The objective of this specification is to establish the general processing requirements for the Aerotherm/Acurex preparation of standard epoxy skinned, Nomex honeycomb core structures.
- 2.0 MATERIALS
- 2.1 F-224 epoxy prepreg cloth (Hexel)
- 2.2 HRH-10, 1/8 in. (0.32 cm) Nomex honeycomb core (Hexcel)
- 2.3 EA 9410 two-part modified paste adhesive (Hysol)
- 2.4 Release paper
- 3.0 EQUIPMENT
- 3.1 Aluminum bag
- 3.2 Press capable of 350°F
- 3.3 Vacuum pump
- 4.0 SAFETY
- 4.1 There are no special safety requirements applicable to this specification.
- 5.0 STORAGE
- 5.1 All raw materials shall be stored in their original containers and shall be protected from contamination.
- 5.2 All materials shall be stored at room temperature unless specified otherwise by the manufacturer.

6.0 PROCEDURE

- 6.1 Curing glass reinforced epoxy skins
 - 6.1.1 Determine the area to be skinned.
 - 6.1.2 Cut this area from the F-224 prepreg cloth, and repeat
 - 6.1.3 Stack these two plies between two plies of Style 51789 nylon peel
 - 6.1.4 Stack these between Teflon release film
 - 6.1.5 Add 1 ply of 120 glass cloth to each side and stack the assembly between steel caul plates inside an aluminum bag.
 - 6.1.6 Under full vacuum raise the temperature from RT to 350°F in 1 hour
 - 6.1.7 Cure at 350°F for 2 hours and cool under vacuum to <150°F
- 6.2 Bonding Skins to Core
 - 6.2.1 Cut size core to be bonded
 - 6.2.2 Cut two 116 E-glass reinforced epoxy laminates to the same dimensions as the core
 - 6.2.3 Mix Parts A and B of EA 9410 and spread on release paper
 - 6.2.4 Move core around adhesive to wet thoroughly the honeycomb edge on one side
 - 6.2.5 Place one skin (laminate) on smooth surface
 - 6.2.6 Place core on top of skin with adhesive impregnated side against the skin
 - 6.2.7 Place caul block on top of core and weight on caul block to achieve 5 psi skin surface area loading
 - 6.2.8 Leave at room temperature overnight
 - 6.2.9 Repeat Steps 6.2.3 through 6.2.8 for other side of core

APPENDIX B

PRELIMINARY PROCESS SPECIFICATIONS FOR PREPARATION AND CURE OF POLYBENZIMIDAZOLE COATED 116 E-GLASS SKINS

- 1.0 SCOPE
- 1.1 The objective of this specification is to establish the general processing requirements for 2 ply 116 glass reinforced polybenzimidazole laminates for use as skins for low-density polybenzimidazole foam.
- 2.0 MATERIALS
- 2.1 PBI 2801 prepolymer
- 2.2 116 E-glass fabric (J. P. Stevens)
- 2.3 104 Glass fabric
- 2.4 TX-1040 Teflon release cloth
- 3.0 EQUIPMENT
- 3.1 Hot melt coater capable of attaining temperatures on the order of 600°F
- 3.2 Press capable of at least 850°F and 200 psi
- 3.3 Aluminum bag
- 4.0 SAFETY
- 4.1 The hot melt coating process and the cure involve the loss of phenol. They should therefore be performed with adequate positive ventilation.
- 5.0 STORAGE
- 5.1 All raw materials shall be stored in their original containers and shall be protected from contamination
 - 5.1.1 PBI 2801 shall be protected from excessive exposure to light, either natural or artificial.

5.1.2 All applicable material shall be stored at room temperature

6.0 PROCEDURE

6.1 Prepregging

- 6.1.1 Determine the area and the weight of 116 E-glass fabric that is to be prepregged
- 6.1.2 Set the temperature of the hot melt coater 20°F to 50°F hotter than the PMT of the PBI 2801 powder to be used.
- 6.1.3 Using standard hot melt coating techniques, pull the coater over the glass fabric.

 Coater should be set to allow for optimum pickup.

6.2 Curing

- 6.2.1 Cut prepreg to the desired dimensions for cure
- 6.2.2 Stack two plies of prepreg together between* plies of TX 1040 Teflon release and 105 bleeder plies. Several laminates may be simultaneously prepared by stacking these arrays together and curing at the same time.
- 6.2.3 Place the assembly in an aluminum foil bag and equip with nitrogen inlet and bubbler. To insure smoothness, the assembly is generally placed between steel or aluminum cauls in the bag.
- 6.2.4 Seal bag and place between platens of press
- 6.2.5 Under contact pressure and positive nitrogen flow, heat to 350°F at 3°F/minute
- 6.2.6 Apply 200 psi and hold 30 minutes
- 6.2.7 Stepwise, cure the part using the following schedule

350-400° 10 min 400° hold 30 min

400-450° 10 min

450° hold 30 min

450-500° 10 min

500° hold 30 min

500-550° 10 min

550° hold 30 min

550-600° 10 min

600° hold 30 min

650-850° 30 min

850° hold 12 hours

Cool under pressure and nitrogen.

^{*}Single ply skins quite porous

- 6.2.8 Cool to ~300°F
- 6.2.9 Remove nitrogen, release pressure and disassemble to remove laminate skins.

APPENDIX C

PRELIMINARY PROCESS SPECIFICATIONS FOR THE PREPARATION OF LOW DENSITY POLYBENZIMIDAZOLE BLOWN FOAM

The preparation of PBI blown foam followed the Preliminary Material, Process and Performance Specifications of Low Density PBI Foam, #2-24804 (DA-6) as modified in Part 1, Section 2 of NAS2-8525 "Study for the Optimization of Thermophysical Properties of Polybenzimidazole Foams."

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APPENDIX D

PRELIMINARY PROCESS SPECIFICATIONS FOR THE BONDING OF GLASS REINFORCED PBI SKINS TO LOW DENSITY PBI BLOWN FOAM WITH PBI 2801 POWDER

- 1.0 SCOPE
- 1.1 The objective of this specification is to establish the general processing requirements for bonding PBI skins to PBI blown foam with PBI powder.
- 2.0 MATERIALS
- 2.1 PBI 2801 powder
- 2.2 PBI skins as prepared (Appendix B)
- 2.3 PBI low density foam as prepared (Appendix C)
- 3.0 EQUIPMENT
- 3.1 Band saw capable of cutting 24 in.
- 3.2 Shims designed to the thickness of the final composite
- 3.3 Aluminum bag
- 3.4 Nitrogen
- 3.5 Press capable of 850°F performance
- 4.0 SAFETY
- 4.1 There are no special safety requirements applicable to this specification.
- 5.0 STORAGE
- 5.1 All materials shall be stored in their original containers and shall be protected from contamination.
 - 5.1.1 PBI 2801 shall be protected from excessive exposure to light, either natural or artificial
 - 5.1.2 All applicable material shall be stored at room temperature

- 6.0 PROCEDURE
- 6.1 Cut the low density PBI blown foam to the desired dimensions with the band saw
- 6.2 Dry foam
- 6.3 Cut the PBI skins to the desired dimensions
- 6.4 Set the temperature of the lower platen of the press 20° to 50° hotter than the PMT of the PBI 2801 powder to be used.
- Place the skins on the hot platen and sprinkle the PBI 2801 (~15 g/ft^2) and allow to melt.
- 6.6 Assemble the following in a nitrogen bag:
 - 1 ply 116 E-glass
 - 1 ply porous Teflon
 - 1 skin coated with PBI 2801 with coated side uppermost
 - PBI foam block
 - 1 skin coated with PBI 2801, with coated side adjacent to foam block
 - 1 ply porous Teflon
 - 1 ply 116 E-glass
- 6.7 Place shims of the proper thickness along side the assembled part
- 6.8 Close the bag and attach to a nitrogen line in one corner and a bubbler in a second corner
- 6.9 Close press to shims and heat from RT 350°F
- 6.10 Hold at $350^{\circ}F$ for 1 hour and then increase heat at a rate of 1-2/3°F/minute until $750^{\circ}F$ is reached
- 6.11 Hold at 750°F for 1 hour then cool under nitrogen
- 6.12 Disassemble bag and remove part
- 6.13 The part is ready for service now or optionally for greater high-temperature performance may be postcured free standing, under nitrogen, in an oven at 850°F for 1 hour

APPENDIX E

PRELIMINARY PROCESS SPECIFICATIONS FOR THE BONDING OF GLASS REINFORCED PBI SKINS TO LOW DENSITY PBI BLOWN FOAM WITH PPQ 401 ADHESIVE

- 1.0 SCOPE
- 1.1 The objective for this specification is to establish the general processing requirements for bonding PBI skins to PBI blown foam with PPQ 401 adhesive
- 2.0 MATERIALS
- 2.1 PPQ 401 adhesive 10% solids in m-cresol
- 2.2 PBI skins as prepared (Appendix B)
- 2.3 PBI low density foam as prepared (Appendix C)
- 3.0 EQUIPMENT
- 3.1 Band saw capable of cutting 24 in.
- 3.2 Shims designed to the thickness of the final composite
- 3.3 Aluminum bag
- 3.4 Nitrogen
- 3.5 Press capable of 700°F performance
- 4.0 SAFETY
- 4.1 Care should be taken to insure that the m-cresol released during the curing segment are expelled from the working area by performing the operation under a forced-draft hood
- 5.0 STORAGE
- 5.1 All materials shall be stored in their original containers and shall be protected from contamination
- 5.2 All applicable material shall be stored at room temperature

- 6.0 PROCEDURE
- 6.1 Cut the low density PBI foam to the desired dimensions with a band saw
- 6.2 Dry the cut foam
- 6.3 Cut the PBI skins to the desired dimensions
- 6.4 Apply a 10 percent solution of PPQ 401 to one side of the PBI skins
- Place the PBI skins on either side of the low density foam so that the PPQ coated sides are adjacent to the foam
- 6.6 Dry assembly by placing in a press, preheated to 400°F, at contact pressure
- 6.7 Remove assembly and place in an aluminum bag between shims
- 6.8 Place the assembly under nitrogen, in a press
- 6.9 Apply 5 psi and heat the press to 500°F; hold at that temperature for 30 min.
- 6.10 Finish the cure using the following schedule:

500°F - 600°F

600°F 30 min

600°F - 700°F

700°F 2 hours

6.11 Cool the assembly in the press until approximately 150°F. Remove part from aluminum bag.

APPENDIX F

PRELIMINARY PROCESS SPECIFICATIONS FOR PREPARATION AND CURE OF KERIMID COATED 116 GLASS SKINS

1.0	SCOPE
1.1	The objective of this specification is to establish the general processing requirements
	for 1 ply 116 glass reinforced Kerimid 601 skins.
2.0	MATERIALS
2.1	Kerimid 601 powder (Rhodia)
2.2	116 E-glass fabric (J. P. Stevens)
2.3	N-methyl-2-pyrrolidinone, NMP (MCB reagent or equivalent)
2.4	F.E.P. Teflon film (Saunders Corporation)
3.0	EQUIPMENT
3.1	Beaker
3.2	Magnetic stirring bar
3.3	Hot plate stirrer (hot plate optional)
3.4	Prepeg drying oven capable of 225°F
3.5	Triple beam pan balance capable of reading 0.1 + 0.01 g and of weighing 100 g
3.6	Aluminum foil bag
3.7	Press autoclave
3.8	Press with upper and lower platens capable of 450°F and at least 100 psi
3.9	Vacuum pump

3.10 Oven capable of 450°F

- 4.0 SAFETY
- 4.1 All operations involving NMP should be performed in a hood or under positive ventilation.
- 5.0 STORAGE
- 5.1 All raw material shall be stored in their original containers and shall be protected from contamination
 - 5.1.1 NMP should be kept dry by appropriate means
 - 5.1.2 All applicable material shall be stored at room temperature
- 6.0 PROCEDURE
- 6.1 Prepregging
 - 6.1.1 Determine the area of 116 E-glass fabric fabric that is to be prepregged.
 - 6.1.2 From the area, calculate the weight of the glass that is to be prepregged
 - 6.1.3 Add 5 percent to this weight. This will be the weight of NMP needed (the 5 percent allocated for waste).
 - 6.1.4 Pour this weight of NMP into an appropriate sized beaker containing the stirring bar (the beaker should have at least twice the volume of the NMP used) and place the vessel on a hot plate stirrer.
 - 6.1.5 With the solution stirring at a moderate rate, slowly add a weight of Kerimid 601 powder equivalent to 7/13 the weight of the NMP. Some heating may be necessary to effect solution within a reasonable period of time.
 - 6.1.6 Coat the glass fabric with the NMP/Kerimid 601 solution prepared above, using any of several known solution prepregging procedures (brush coating the solution was found to be most effective). For a 35 percent solids pickup, all of the solution should be used allowing for 5 percent waste.
 - 6.1.7 Place the prepreg into the prepreg drying oven, preset at 225°F and dry for 50 minutes.
 - 6.1.8 Remove the prepreg from the film and hang the prepreg in the oven so both sides are free. Dry an additional 5 minutes in this form.
 - 6.1.9 At this point the prepreg can be removed and stored. It can be stored indefinitely at room temperature with no special precautions needed other than it be



free from possible contamination. In this form the prepreg should contain 35 ± 3 percent resin and 3 ± 1 percent volatiles (NMP).

6.2 Curing

- 6.2.1 Cut prepreg to the desired dimensions for cure
- 6.2.2 Stack prepreg between two plays of FEP Teflon film and this between two aluminum cauls
- 6.2.3 Place the assembly in an aluminum foil bag inserted in a press autoclave
- 6.2.4 Place press autoclave in a suitable press
- 6.2.5 Attach a vacuum pump to the press autoclave
- 6.2.6 Under full vacuum and 100 psi platen pressure, heat the part to 360°F and hold for 2 hours
- 6.2.7 Under pressure and vacuum, allow to cool to 150°F
- 6.2.8 Remove vacuum, release pressure and when cool enough, disassembly autoclave to remove skins
- 6.2.9 Cure skins an additional 16 hours unrestrained in oven set at 450°I

APPENDIX G

PRELIMINARY PROCESS SPECIFICATIONS FOR P-NASA FOAM FILLED 3/8" KERIMID CORE FOAM COMPOSITES

- 1.0 SCOPE
- 1.1 These specifications are based on the preliminary specification, Polyquinoxaline Foam Composite (Rough Draft #2) by W. J. Gilwee. They are modified with respect to the larger 3/8 in. honeycomb cell size to be filled and to the method of bonding the Kerimid skins.
- 2.0 MATERIALS
- 2.1 Bismalimid honeycomb core 3/8 in. cell diameter (Hexcel)
- 2.2 Kerimid/116 E=glass skins (Appendix F)
- 2.3 BR-34 primer (American Cyanimide)
- 2.4 FM-34 adhesive (American Cyanimide)
- 2.5 Celluose Nitrate, 18 percent solids in MEK (DuPont)
- 2.6 Ammonimum 2-amino-5-nitrobenzene sulfonate (p-NASA salt) (AVCO)
- 2.7 Toluene
- 2.8 Ethyl acetate
- 3.0 EQUIPMENT
- 3.1 Paint shaker, closed container and ceramic balls for achieving emulsion
- 3.2 Drying oven capable of 100°C
- 3.3 Second oven capable of 280°C
- 3.4 12 mesh wire screen
- 3.5 Flat platen press capable of 550°F and 40 psi

- 4.0 SAFETY
- 4.1 Adequate ventilation \underline{must} be employed in the dipping, drying and foaming operations involving the p-NASA salt
- 4.2 If an oven is used in the foaming operation, it must be explosion-proof
- 5.0 STORAGE
- 5.1 All raw materials shall be stored in their original containers and shall be protected from contamination.
 - 5.1.1 Solvents shall be stored in accordance with rules and regulations regarding storage of such
 - 5.1.2 Celluose nitrate shall not be allowed to dry out
 - 5.1.3 Kerimid skins and p-NASA foam filled honeycomb core shall be dried prior to use
- 6.0 PROCEDURE
- 6.1 Description of Polyquinoxaline Foam
 - 6.1.1 Coating: 41 percent nitrocellulose solution, 5 sec. type, 18 percent solids MEK
 - 23 percent p-NASA salt ("para nitroaniline ortho sulfonic acid, ammonium salt")
 - 18 percent toluene
 - 18 percent ethyl acetate
 - 6.1.2 Above material mixed in 1 gallon can and mixed on a paint shaker for 30 min. with 6-10, 1/2-3/4 in. ceramic balls.
 - 6.1.3 The coating was then dip coated on honeycomb (4 g/sq. in. wet weight) and dried at 1 hr at 100°C. The dry coating weight should be 1.2 to 1.5 g/sq. in.
 - 6.1.4 Preheat flat platen press to 280°C. Note: Steps 6.1.4.1 to 6.1.4.3 must be performed in a hood or spray booth.
 - 6.1.4.1 Place wire screen (12 mesh) on top and bottom and two layers of glass bleeder cloth on top and bottom. Sandwich between 5 mil Teflon film and aluminum plates (0.125 in.) and place in preheated press at contact pressure, or:

- 6.1.4.2 Place wire screen (12 mesh) top and bottom of painted core. A performated metal plate (10 holes/in., 0.030 in. thick) is then placed on top, metal rods (0.375 in. diameter) are then placed at inch intervals on top and the entire assembly is placed between heated platens; or:
- 6.1.4.3 The assembly in 6.1.4.2 is restrained and clamped in an open metal frame with metal cross members. A propane torch is then used to activate the polyquinoxaline foam. The foaming action is self-propagating.
- 6.1.5 Intumescence will occur in approximately 2 min.
- 6.1.6 After 5 min. remove the screens
- 6.1.7 Foam density 1.5 to 2.0 lb/ft^3
- 6.1.8 For 3/8 in. cell honeycomb core a repeat of Steps 6.1.3 to 6.1.6 was sometimes necessary as the original foaming would not fill the center of the cells.
- 6.2 Adhesive Bonding Face Sheet to Core
 - 6.2.1 Bristle brush the foam/core to provide 1/32 in. fillet area on honeycomb
 - 6.2.2 Coat both sides of honeycomb with BR-34 adhesive
 - 6.2.3 Air dry 1 hour at RT and finally at 220°F for 1/2 hour
 - 6.2.4 Lightly sand one side of Kerimid skin after cleaning with methylene chloride
 - 6.2.5 Place FM-34 adhesive on core area and skins on FM-34 adhesive with sanded side adjacent to adhesive; or:
 - 6.2.6 Place skins on core area with sanded side adjacent to primer
 - 6.2.7 Place assembly in aluminum bag with 2 plys 120 glass and 2 plys 181 glass between the assembly and the inner walls of the bag
 - 6.2.8 Seal bag and allow for nitrogen inlet and exit
 - 6.2.9 Place between platens of press and while applying 40 psi, heat from RT to 350°F
 - 6.2.10 Hold at 350°F for 45 min; heat to 550°F over 2 hours, and hold at 550°F for 2 hours
 - 6.2.11 Cool under pressure and remove part
 - 6.2.11 Trim to final dimensions

- 6.3 Bonding Decorative Face Sheet to Composite after Postcure
 - 6.3.1 Adhesive made up as follows:

Epon 828 50 parts
Chlorendic anhydride 48 parts
DMP 30 1 part
MEK 21 parts

- 6.3.2 The above adhesive is coated on panel after postcure. The wet weight of adhesive is to be 0.22 g/in^2 .
- 6.3.3 Air dry 10 minutes and oven dry 10 minutes at 100° C. The dry weight of adhesive is 0.15 g/in^2 .
- 6.3.4 Place phenylphthalein polycarbonate skins on tacky surface and place between porous Teflon film, 6 plays 120 glass and 1 ply alpha celluose and hold in press at 25 psi at 302°F.